

# **An End Station for Intermediate X-Ray Absorption Spectroscopy of Biological Samples in a Controlled Environment**

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X-ray absorption spectroscopy (XAS) is an element-specific technique with widespread applications in structural biology. Most notably, it has been used in the hard x-ray region ( $> 6$  keV) to probe metal centers in metalloproteins. In contrast, investigations of low-Z atoms (e.g., S, Cl, Ca) within proteins and other biological samples have been scarce. Such experiments require the use of a cryostat with vacuum insulation since low temperatures are required for protein samples to minimize radiation damage and for data collection in the EXAFS region. Existing instrumentation does not allow substantial transmittance of intermediate x-rays through the windows to reach the sample which inhibits adequate flux of fluorescent x-rays to reach the detector.

A new end station on Beamline 9.3.1 at the ALS has been designed and built for such XAS experiments in the 2-6 keV region for solid, liquid or solution samples under selectable temperature and pressure. The sample chamber contains provisions for a liquid helium cryostat such that samples may be cooled to any temperature down to 10 K and maintained at 1 atm pressure. The sample and exchange gas are housed within an inner sample chamber with minimally attenuating windows that are able to withstand a 1 atm pressure differential (0.25 mil Mylar/Kapton or 38 mm Be). The sample surface is positioned at a selectable angle (e.g.,  $45^\circ$ ) to the incident beam, and the fluorescent signal is collected by a Si photodiode housed within the inner sample chamber. The internal detector is situated 1 cm away from the sample which allows collection of a large solid angle of fluorescent x-rays. There are also provisions for an externally mounted Ge energy-resolving detector. A retractable Si

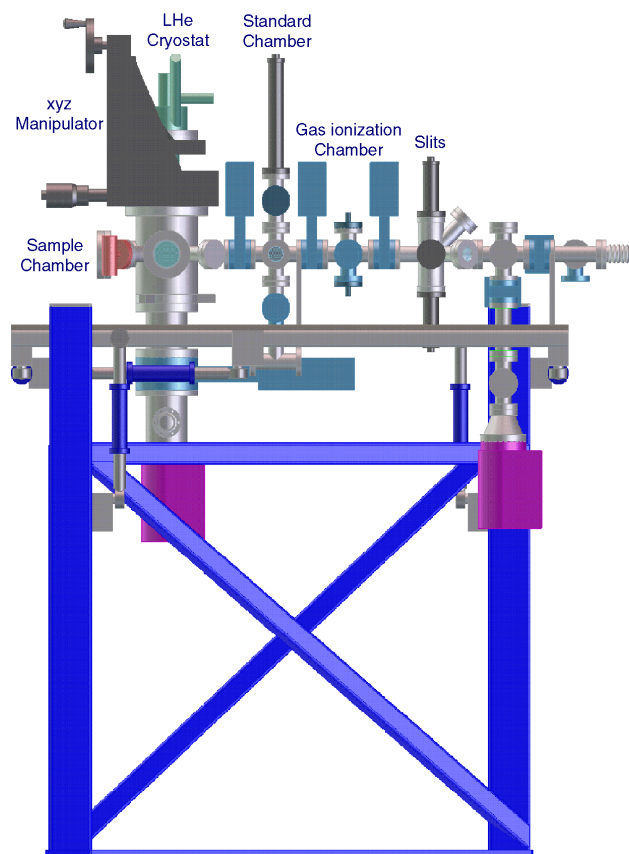


Figure 1. Beamline 9.3.1 end station.

photodiode downstream from the internal sample chamber may be used for transmittance experiments or sample positioning.

Upstream from the sample chamber is a smaller chamber which houses a grid for monitoring incident beam intensity and standard samples for energy calibration. An ion chamber enclosed by 2000 Å parylene-N windows may also be used for incident flux/energy calibration.

The new instrumentation is ready for beam immediately and will provide a general purpose XAS end station for samples in a controlled environment. Initially it will be suited for protein samples, but modifications are being planned for applications in material science.

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